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AMMONIA DETERMINATION IN URANIUM PROCESS SOLUTIONS

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ABSTRACT

The Division of Nuclear Fuel Cycle of the International Atomic Energy Agency requested the participation of CANMET in the preparation of a manual of analytical methods for the uranium industry. The staff of the Chemical Laboratory has responded to this request and will issue several reports describing current methodology and laboratory practice. This report describes the determination of ammonia by steam distillation and titration with standard hydrochloric acid.

Keywords: Uranium process solutions, ammonia, steam distillation, acidimetric determination.

DÉTERMINATION DE L'AMMONIAC DANS LES SOLUTIONS DE TRAITEMENT DE L'URANIUM

par

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La division du cycle du combustible nucléaire de l'Agence internationale de l'énergie atomique a demandé la participation du CANMET pour la préparation d'un manuel sur les méthodes analytiques utilisées dans l'industrie de l'uranium. Le personnel du laboratoire de chimie s'est appliqué à cette tâche et il publiera plusieurs rapports decrivant la méthodologie courante et les pratiques habituelles dans ce domaine. Le présent rapport décrit la détermination à l'ammoniac par distillation à la vapeur et par titrage avec de l'acide chlorhydrique standard.

Mots-clés: solutions de traitement de l'uranium, ammoniac, distillation à la vapeur, détermination acidimétrique

INTRODUCTION

This method is applicable to the determination of ammonia nitrogen in samples from uranium process circuits.

The ammonia is released by steam distillation from strong alkaline solution, and is absorbed by a solution of boric acid. The ammonia is determined acidimetrically with standard hydrochloric acid, using methyl red as an indicator.

INTERFERENCES

Some organic compounds such as amines and amides may give ammonia in hot alkaline solutions or be volatilized and give rise to errors. Urea and cyanates will hydrolyze on distillations at a high pH.

RANGE

This method is suitable for samples containing ca. 0.1 mg/L and higher concentrations of ammonia.

EQUIPMENT

Automatic steam distillation systems are available from Brinkman Instruments Co., Rexdale, Ont., Canada (Bucki-Brinkman model 320) and Tecator Inc., Boulder, Co. USA (Tecator model 1003).

REAGENTS

All solutions are prepared from distilled water purified with a Milli-Q (Millipore) system.

Ammonia Standard Solution (0.50 $Mol \cdot L^{-1}$) - Dry reagent ammonium sulphate ($(NH_4)_2SO_4$) for 1 h at 100°C. Accurately weigh 1.607 g and dissolve in water. Dilute to 1 litre in a volumetric flask.

Boric Acid Solution (3% W/V) - Dissolve 30 g of boric acid (H_3BO_3) in water and dilute to 1 litre.

Hydrochloric Acid, Standard Solution (0.05) - Dilute 100 mL of 1.0 M HCl to 2 litres. Standardize in accordance to Procedure, and calculate mg of ammonia equivalent to 1 mL of standard HCl solution.

Indicator Solution (1 g/L) - Dissolve 0.1 g of methyl red in 100 mL of ethanol (95%).

Sodium Hydroxide Solution (25% W/V) - Dissolve 250 g of NaOH in water and dilute to 1 litre.

CALIBRATION

Transfer 5 mL sample of standard ammonium solution to distillation tube and distill the ammonia according to Procedure. Titrate with 0.05 $Mol \cdot L^{-1}$ HCl to methyl-red end point. Calculate mg of ammonia equivalent to 1.00 mL of HCl.

PROCEDURE

Refer to manufacture's manual for operation of distillation apparatus. Distill at least two water samples before commencing with samples.

Transfer a sample containing not more than 1.5 mg of ammonia nitrogen to distillation tube. Set tube into distillation apparatus. Add 50 mL of boric acid solution to collection flash. Add water to sample to adjust volume in distillation tube to about 70 mL, then add 30 mL of NaOH solution. Start steam distillation and collect about 100 mL of distillate. Titrate with standardized HCl solution to methyl-red end point.

CALCULATION

Calculate the ammonia concentration in mg/litre of ammonia in original sample as follows:

ammonia nitrogen, mg/litre

$$= A \times E \times \frac{1000}{V}$$

where:

A = mL of HCl

E = mg NH^{3+} per mL of HCl titrant.

V = sample volume

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